

JAPANESE [JP,2001-055672,A]

CLAIMS **DETAILED DESCRIPTION** **TECHNICAL FIELD** **PRIOR ART** **EFFECT**
OF THE INVENTION **TECHNICAL PROBLEM** **MEANS** **EXAMPLE**

[Translation done.]

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Field of the Invention]This invention belongs to the technical field for coloring textiles, and relates to the new art in which an animal fiber etc. can be colored without using a color especially.

[0002]

[Description of the Prior Art]In order to color various kinds of textiles, dyeing using a color is chiefly performed from before. For example, dyeing of silk and the wool which are animal fibers is performed using acid dye, a direct color, etc. The side chain of the amino acid in which this dyeing mechanism constitutes the animal fiber (protein) (- sulphydryl group etc.), Or the amino group (-NH₂) and carboxylic acid group (-COOH) which are located in a proteinic end are made to ionize, and a color is made to adhere, when these carry out an ionic bond to a color with a counter ion. There is also a thing using the physical adsorption produced although a color enters into textiles.

[0003]It has a color, though these colors are natural. That is, dyeing of the conventional textiles twists a compound (color) with a color to a certain thing [making it adhere to textiles using chemical or a physical interaction, and coloring these textiles]. And various chemical dye which can be dyed with such a mechanism is developed. However, when a color is absorbed by the human body in recent years, some what has an adverse effect on the body, and things which produce environmental deterioration from wastewater are reported, and manufacture and import are forbidden to some them. For this reason, although the tendency which shifts to natural dye as much as possible is seen, generally, a natural color has the low fastness by wash, friction, sweat, etc., and development of new art is needed.

[0004]

[Problem(s) to be Solved by the Invention]The purpose of this invention is to provide the new art outstanding also in fastness etc. moreover, without using the

color which may have an adverse effect on a human body or environment in coloring an animal fiber etc.

[0005]

[Means for Solving the Problem]this invention person thought out textiles coloring art new type of using the coloring reaction, paying attention to tryptophan which is one of the amino acid.

[0006]That is, this invention first provides a coloring method of textiles including a process of processing textiles which contained tryptophan on a target by birth or into which tryptophan was introduced from the exterior with acid and an aldehyde compound, as a basic invention.

[0007]This invention is suitable for a target by birth textiles which comprise protein containing tryptophan, and coloring an animal fiber especially, and is applied as a coloring method of an animal fiber including a process of processing an animal fiber with acid and an aldehyde compound. Especially a method of this invention is applied, a desirable animal fiber is silk or wool.

[0008]Colorant of an animal fiber characterized by including acid and an aldehyde compound from another viewpoint also provides this invention.

[0009]

[Embodiment of the Invention]The coloring art of this invention gives a color to the textiles itself using the coloring reaction of tryptophan, and differs from the old textiles dyeing machine style based on making a color adhere to textiles fundamentally.

[0010]About tryptophan, a coloring reaction called the Hopkins Kohl reaction (Hopkins-Cole Reaction) and a neubauer-rhode's reaction (Neubauer-Rhode Reaction) is known well. If glyoxylic acid and sulfuric acid are added to the acetic acid solution of tryptophan, a red purple color will present the former, The latter is a reaction which a red purple color - a blue-purple color present, when dimethylbenzaldehyde is added to the concentrated-hydrochloric-acid solution of tryptophan, and it is used for a fixed quantity of the sample in which all contain tryptophan, and detection.

[0011]However, there is no way of thinking of using the coloring reaction of tryptophan about coloring of textiles conventionally, and it was considered rather that such coloration was what is a fault and should be avoided. For example, although yellow change is made an issue of in silk or wool, it is thought that the cause is in existence of tryptophan.

[0012]If this invention person processes the textiles which have tryptophan with acid and an aldehyde compound as a result of advancing research on the basis of the coloring reaction (color reaction) of the above tryptophan, Coloring of textiles is attained without completely using a color, The new textiles coloring method which can also adjust the concentration of a color was drawn by changing the kind and combination of these acid and an aldehyde compound by acquiring various colors and adjusting the quantity of these acid and/or an aldehyde compound.

[0013]Although the principle of this invention is applied to coloring the textiles which have tryptophan on a target or and extrinsically by birth, it is suitable to color a target especially the textiles which have tryptophan, i.e., a protein fiber, by birth. As a protein fiber applied, an animal fiber especially wool, or silk is common. As a protein fiber, the reproduction protein fiber which uses casein, zein, peanut protein, etc. other than an animal fiber as a raw material is also mentioned, and this invention can be applied also to coloring of these textiles.

[0014]textiles [like the animal fiber (protein fiber) on **] whose method of this invention is and which contain tryptophan on a target by birth -- in addition, it is applicable also to coloring the textiles which have tryptophan extrinsically, i.e., the textiles into which tryptophan was introduced from the exterior. For example,

according to this invention, a cotton fiber can also be colored by introducing and combining tryptophan with the hydroxyl group of the cellulose which constitutes cotton by a coupling reaction.

[0015]Although the acid used in this invention belongs to strong acid, it must not dissolve textiles. Although the acid used at the Hopkins Kohl reaction using the neubauer-rhode's reaction and glyoxylic acid using this point and 4-dimethylaminobenzaldehyde is a mixture of concentrated hydrochloric acid, glacial acetic acid, and concentrated sulfuric acid, respectively, These are not preferred at that (textiles will dissolve) which loses the endurance of textiles. It is preferred to dilute and use strong acid (organic acid or inorganic acid is not asked), for example, trifluoroacetic acid, dichloroacetic acid, chloride, etc. with water or weak acid (for example, acetic acid) in this invention in this way.

[0016]Although the aldehyde compound used in this invention is chosen combining acid according to the color for which it asks, generally, its aromatic system aldehyde compound is preferred, and especially its aldehyde compound that comprises benzaldehyde as a basic skeleton especially is preferred. If the glyoxylic acid which is this point and the aliphatic series system aldehyde compound used at the Hopkins Kohl reaction is used, it is found out that silk and wool can hardly be colored.

[0017]If this invention is followed, various colors can be acquired by changing the combination of the above aldehyde compounds and acid. For example, when trifluoroacetic acid is used as acid, in coloring of silk as an aldehyde compound, If benzaldehyde, 4-hydroxybenzaldehyde, 3,4-dihydroxybenzaldehyde (PUROTO catechu aldehyde), or 9-antha aldehyde is used, silk can be colored green, a red purple color, purple, or the color of a yellow system, respectively. When coloring silk, using 4-hydroxybenzaldehyde as an aldehyde compound, a red purple color, reddish brown, or pink can be obtained, respectively by using trifluoroacetic acid, dichloroacetic acid, or HBr/acetic acid as acid.

[0018]If this invention is followed, these textiles can be colored only by the textiles which have tryptophan being immersed into the mixed solution of acid which was mentioned above, and an aldehyde compound, and this coloring hardly changing with wash, friction, sweat, etc., but having high fastness is checked.

[0019]On the occasion of coloring processing, as mentioned above, generally a predetermined aldehyde compound is added to the A acid solution which diluted and prepared strong acid with acetic acid etc., and acid / aldehyde compound mixed solution is prepared. The ratio in particular of acid and an aldehyde compound is not limited, and is defined according to the grade of the shade of a desired color.

[0020]Thus, the mixed solution of the obtained acid and an aldehyde compound is made to immerse the 1st to about 2nd generally, shaking predetermined textiles preferably. What is necessary is just to air-dry at ordinary temperature, after a detergent (generally water or water / methanol mixed liquor) suitable as post-processing will wash, if desired coloring is attained by immersion.

[0021]

[Example]Hereafter, in order to carry out the feature of this invention for whether being ** further, an animal fiber is colored according to this invention, and the example which measured the fastness is shown, but this invention is not restricted by these examples. The coloring obtained in each example is displayed by the thing to depend on macroscopic observation, and the L*a*b* diagram.

The latter was measured by SpectroColor Meter SE 2000 of JEOL Industry.

[0022]Wash, friction, and the fastness test to sweat were done according to JIS L 0844, JIS L 0849, and JIS L 0848 of Japanese Standards Association, respectively. Although the details of each test method are as regulation to JIS, it will be as follows if it sketches.

[0023]Two kinds of undyed cloth for staining (the same kind as a sample and

cotton) are sewn on a sample, a specimen is produced, and it puts into an examination bottle with the soap solution to which this was specified, and washes at 50 ** for 30 minutes by wash examination (JIS L 0844) using a regular washing machine.

[0024]A rubbing test (JIS L 0849) rubs a specimen and the white cheesecloth for friction of each other 100 times at a fixed speed using a frictional testing machine. In the 2 passage of dryness and a damp or wet condition, a white cheesecloth carries out in this examination.

[0025]The sweat test (JIS L 0848) is immersed in two kinds of artificial sweat solution, acid and alkali, which sewed two kinds of undyed cloth for staining (the same kind as a sample, and cotton) on the sample, produced the specimen, and produced this by the regular method at ordinary temperature for 30 minutes. Next, it holds with a 37 ** dryer for 4 hours, inserting a sample into a rigid plastic board and putting a fixed pressure.

[0026]The contamination result in these fastness tests was judged with the gray scale for contamination (L 0805) to be that of a standard illuminant. The gray scale for contamination serves as a standard which judges the grade of the contamination produced on the white cloth by visual appreciation, and is divided to the 1 to 5th class in regular color difference. Here, it means not being polluted, whenever nine color charts, such as the 1st class, the one to 2nd class, and the 3rd [-] the class [2nd] two class, show, the 1st class is polluted most and a series increases.

[0027]Example 1: The silk (2-2) and the wool of the JIS standard adjacent fabric for staining of colour fastness test (JIS L 0803 conformity) of Japanese Standards

Association were used for the sample of the coloring animal fiber of the animal fiber using trifluoroacetic acid and 4-hydroxybenzaldehyde as 25x22 cm.20 ml of trifluoroacetic acid (Watanabe Chemical industry) was diluted with 40 ml of acetic acid (best of Kishida Chemical Industry) among a 110-ml sample tubing, and 1 g of 4-hydroxybenzaldehyde (best of Wako Pure Chemical Industries, Ltd.) was added. The undyed cloth for staining of silk was put into this mixed solution, and it shook for 40 ** and two days with the shaker. Post-processing was washed using water and methanol and was dried in the room temperature air. As a result, as shown in Table 1, the undyed cloth for staining of silk was colored the red purple color ($L^*a^*b^*=66.03, 18.15, 2.36$). When similarly carried out about wool, it was colored the dark red purple color ($L^*a^*b^*=61.58, 14.51, 2.69$).

[0028]Dark color-ization of coloring of textiles was performed by increasing the quantity of acid and an aldehyde compound as follows. First, the trifluoroacetic acid which is acid was 20 ml like the above, increased 4-hydroxybenzaldehyde which is an aldehyde compound to 2 g, and colored silk. Subsequent conditions and processing were performed like the above. $L^*a^*b^*$ of silk was set to 62.79, 18.27, and -1.44, L^* showing a shade decreased by 3.24, and the result checked that-izing could be carried out [a dark color] (Table 1). The place which colored 4-hydroxybenzaldehyde by increasing the quantity of 1 g and trifluoroacetic acid with 30 ml, $L^*a^*b^*$ of silk was set to 57.37, 16.50, and -5.11, L^* decreased by 8.66 reduction, wool decreased 0.69 times by 60.89, 17.86, and 4.98, and it checked that-izing could be carried out [a dark color] by increasing the quantity of acid and an aldehyde compound (Table 1).

[0029]The examination on contamination of these wash, friction, and a color-fastness-to-perspiration examination was done. As a result, the 4th more than class was obtained in all, and fastness became clear [a high thing]. The result of this fastness test is shown in Table 2.

[0030]Example 2: The sample of the coloring animal fiber of the animal fiber using trifluoroacetic acid and benzaldehyde is the same as that of Example 1.20 ml of trifluoroacetic acid was diluted with 40 ml of acetic acid among a 110-ml sample

tubing, and 1 ml of benzaldehyde (best of Wako Pure Chemical Industries, Ltd.) was added. The undyed cloth for staining of silk was put into this mixed solution, and it shook for 40 ** and two days with the shaker. Post-processing was washed using water and methanol and was dried in the room temperature air. As a result, as shown in Table 1, the undyed cloth for staining of silk was colored green ($L^*a^*b^*=81.57, -4.36, 17.18$). When similarly carried out about wool, it was colored a yellowish brown color ($L^*a^*b^*=88.27, -3.77, 20.34$). This dark color-ization was performed by increasing the quantity of trifluoroacetic acid for benzaldehyde to 30 ml at 1 ml. The result checked that $L^*a^*b^*$ of silk is set to 71.26, -6.76, and 19.36, L^* decreased by 10.31 reduction, wool decreased 1.89 times by 86.38, -2.15, and 22.64, and a dark color could be carried out also here. Also in the examination on contamination of wash, friction, and a color-fastness-to-perspiration examination, all brought a result of the 4th more than class (Table 2).

[0031]Example 3: The sample of the coloring animal fiber of trifluoroacetic acid and the animal fiber using 9-antra aldehyde is the same as that of Example 1.20 ml of trifluoroacetic acid was diluted with 40 ml of acetic acid among a 110-ml sample tubing, and 1 g of 9-antra aldehyde (Tokyo Kasei Kogyo Co., Ltd.) was added. Undyed cloth for staining was put into this mixed solution, and it shook for 40 ** and two days with the shaker. Post-processing was washed using water and methanol and was dried in the room temperature air. As a result, as shown in Table 1, the undyed cloth for staining of silk was colored light yellow, and wool was colored yellow. The result of the examination on contamination of wash performed like Example 1, friction, and a color-fastness-to-perspiration examination is shown in Table 2.

[0032]Example 4: The sample of the coloring animal fiber of trifluoroacetic acid and the animal fiber using PUROTO catechu aldehyde is the same as that of Example 1.20 ml of trifluoroacetic acid was diluted with 40 ml of acetic acid among a 110-ml sample tubing, and 1 g of PUROTO catechu aldehyde (3,4-dihydroxybenzaldehyde) (best of Wako Pure Chemical Industries, Ltd.) was added. Undyed cloth for staining was put into this mixed solution, and it shook for 40 ** and two days with the shaker. Post-processing was washed using water and methanol and was dried in the room temperature air. As a result, as shown in Table 1, the undyed cloth for staining of silk was colored purple, and wool was colored dark purple. Also in wash, friction, and the examination on contamination of color fastness to perspiration, all brought a result of the 4th more than class (Table 2).

[0033]As mentioned above, even if it used the same acid, it became clear from the result of Examples 1-4 that a color can be changed by changing the kind of aldehyde compound (refer to Table 1).

[0034]The sample of the coloring animal fiber of the animal fiber using 5:30% of example HBr / acetic acid, and 4-hydroxybenzaldehyde is the same as that of Example 1.20 ml of 30%HBr / acetic acid (Watanabe Chemical industry) were diluted with 60 ml of acetic acid among a 110-ml sample tubing, and 4-hydroxybenzaldehyde 1g was added. Undyed cloth for staining was put into this mixed solution, and it shook for two days in a room temperature with the shaker. As a result, as shown in Table 1, the undyed cloth for staining of silk was colored pink, and wool was colored brown. As for all examinations on contamination of wash, friction, and a color-fastness-to-perspiration examination, the result of the 4th more than class was obtained (Table 2).

[0035]Example 6: The sample of the coloring animal fiber of the animal fiber using dichloroacetic acid and 4-hydroxybenzaldehyde is the same as that of Example 1.40 ml of dichloroacetic acid (Tokyo Kasei Kogyo Co., Ltd.) was diluted with 30 ml of acetic acid among a 110-ml sample tubing, and 4-hydroxybenzaldehyde 1g was added. Undyed cloth for staining was put into this mixed solution, and it shook for

40 ** and two days with the shaker. As a result, as shown in Table 1, silk was colored reddish brown and wool was colored brown. All examinations on contamination of wash, friction, and a color-fastness-to-perspiration examination were the 4th more than class (Table 2).

[0036]Even if it used the same aldehyde compound, the color was able to be changed from the result of the above Examples 1, 5, and 6 by changing the kind of acid (Table 1).

[0037]Example 7: The sample of the coloring animal fiber of the animal fiber using dichloroacetic acid and benzaldehyde is the same as that of Example 1.40 ml of dichloroacetic acid was diluted with 30 ml of acetic acid among a 110-ml sample tubing, and 1 ml of benzaldehyde was added. Undyed cloth for staining was put into this mixed solution, and it shook for 40 ** and two days with the shaker. As a result, as shown in Table 1, silk was colored yellowish green and wool was colored yellow. Also in the examination on contamination of wash, friction, and a color-fastness-to-perspiration examination, all brought a result of the 4th more than class (Table 2).

[0038]Even if it used the same aldehyde compound, it was able to be made to change also from the result of the above Examples 2 and 7 by changing the kind of acid (Table 1).

[0039]Example 8: The sample of the coloring animal fiber of dichloroacetic acid and the animal fiber using PUROTO catechu aldehyde is the same as that of Example 1.40 ml of dichloroacetic acid was diluted with 30 ml of acetic acid among a 110-ml sample tubing, and 1 g of PUROTO catechu aldehyde was added. Undyed cloth for staining was put into this mixed solution, and it shook for 40 ** and two days with the shaker. As a result, as shown in Table 1, silk was colored purple and wool was colored brown. Also in the examination on contamination of wash, friction, and a color-fastness-to-perspiration examination, all brought a result of the 4th more than class (Table 2).

[0040]Example 9: The sample of the coloring animal fiber of the animal fiber using chloride and PUROTO catechu aldehyde is the same as that of Example 1.50 ml of 2N chloride (best of Kishida Chemical Industry) was diluted with 10 ml of acetic acid among a 110-ml sample tubing, and 1 g of PUROTO catechu aldehyde was added. Undyed cloth for staining was put into this mixed solution, and it shook for a room temperature and two days with the shaker. As a result, as shown in Table 1, silk was colored pink and wool was colored reddish brown. Also in the examination on contamination of wash, friction, and a color-fastness-to-perspiration examination, all brought a result of the 4th more than class (Table 2).

[0041]Even if it used the same aldehyde compound, the color was able to be changed also from the result of the above Examples 4, 8, and 9 by changing the kind of acid (Table 1).

[0042]Example 10: The sample of the coloring animal fiber of the animal fiber using trifluoroacetic acid and 4-dimethylaminobenzaldehyde is the same as that of Example 1.20 ml of trifluoroacetic acid (Watanabe Chemical industry) was diluted with 40 ml of acetic acid (best of Kishida Chemical Industry) among a 110-ml sample tubing, and 1 g of 4-dimethylaminobenzaldehyde (best of Kishida Chemical Industry) was added. The undyed cloth for staining of silk was put into this mixed solution, and it shook for 40 ** and two days with the shaker. Post-processing was washed using water and methanol and was dried in the room temperature air. As a result, as shown in Table 1, the undyed cloth for staining of silk became light brown ($L^*a^*b^*=86.86, -1.45, 14.43$), and was only colored slightly. When similarly carried out about wool, it was colored green ($L^*a^*b^*=57.49, -3.64, 5.42$) here. The examination on contamination of wash, friction, and a color-fastness-to-perspiration examination was done also about these. Woolen fastness was lower than each of above-mentioned examples. Although the fastness of silk is high, this will be

because coloring hardly arose from the first.

[0043] Example 11: The sample of the coloring animal fiber of the animal fiber using trifluoroacetic acid and glyoxylic acid is the same as that of Example 1.20 ml of trifluoroacetic acid was diluted with 40 ml of acetic acid among a 110-ml sample tubing, and 1 g of glyoxylic acid (best of Kishida Chemical Industry) was added. Undyed cloth for staining was put into this mixed solution, and it shook for 40 ** and two days with the shaker. Post-processing was washed using water and methanol and was dried in the room temperature air. As a result, as shown in Table 1, silk was hardly colored ($L^*a^*b^*=96.30, -2.86, 7.65$). It colored slightly and wool became light yellow ($L^*a^*b^*=92.44, -4.50, 24.03$). The result related with contamination of a fastness test is shown in Table 2. Although fastness is high, this will be for coloring to hardly arise.

[0044]

[Effect of the Invention] Since textiles, such as silk and wool, can be colored without completely using a color if the coloring method or colorant of this invention is used, it does not have an adverse effect of the body and the problem of the environmental pollution by an effluent is not produced, either. If this invention is followed, the processing under ordinary temperature or the low temperature near the ordinary temperature can color a variety only by changing the combination and quantity (concentration) of acid and an aldehyde compound, yellowing of silk and the wool from which tryptophan is considered to be the cause is also pressed down, and operation will be cheap also simply and in cost and will end.

[0045]

[Table 1]

動物纖維の着色結果

	纖維	酸	アルデヒト化合物	色	$L^*a^*b^*$ イグ*ラム
例 1	絹 羊毛 20ml	トリフルオロ酢酸 20ml	4-ヒドロキシベンズアルデヒト	1g 赤紫 濃赤紫	66.03, 18.15, 2.36 61.58, 14.51, 2.69
	絹 羊毛 20ml	トリフルオロ酢酸 20ml	4-ヒドロキシベンズアルデヒト	2g 赤紫	62.79, 18.27, -1.44
	絹 羊毛 30ml	トリフルオロ酢酸 30ml	4-ヒドロキシベンズアルデヒト	1g 濃赤紫 濃赤紫	57.37, 16.50, -5.11 60.89, 17.86, 4.98
例 2	絹 羊毛 20ml	トリフルオロ酢酸 20ml	ベンズアルデヒト	1ml 黄褐	81.57, -4.36, 17.18 88.27, -3.77, 20.34
	絹 羊毛 30ml	トリフルオロ酢酸 30ml	ベンズアルデヒト	1ml 黄褐	71.26, -6.76, 19.36 86.38, -2.15, 22.64
例 3	絹 羊毛 20ml	トリフルオロ酢酸 20ml	9-アントラルデヒト	1g 淡黄 黄	95.81, -6.41, 16.29 87.89, 15.57, 7.61
例 4	絹 羊毛 20ml	トリフルオロ酢酸 20ml	アロトカテキュアルデヒト	1g 濃紫	65.87, 17.14, -4.91 60.22, 15.57, 7.61
例 5	絹 羊毛 20ml	30%HB r / 醋酸 20ml	4-ヒドロキシベンズアルデヒト	1g ピンク 茶	85.57, 8.49, 4.87 68.75, 8.62, 15.14
例 6	絹 羊毛 40ml	ジクロ酢酸 40ml	4-ヒドロキシベンズアルデヒト	1g 赤褐 茶	78.38, 7.87, 9.82 72.18, 6.82, 18.25
例 7	絹 羊毛 40ml	ジクロ酢酸 40ml	ベンズアルデヒト	1ml 黄緑 黄	77.38, -4.44, 35.21 85.32, -3.28, 20.81
例 8	絹 羊毛 40ml	ジクロ酢酸 40ml	アロトカテキュアルデヒト	1g 茶	71.62, 11.25, -2.05 73.10, 5.40, 14.78
例 9	絹 羊毛	塩酸	アロトカテキュアルデヒト	1g ピンク 赤褐	85.70, 9.10, 4.52 76.63, 12.36, 12.86
例 10	絹 羊毛 20ml	トリフルオロ酢酸 20ml	4-シメタカルミノベンズアルデヒト	1g 淡褐色 綠	86.86, -1.45, 14.43 57.49, -3.64, 5.42
例 11	絹 羊毛 20ml	トリフルオロ酢酸 20ml	ケリオキシ酸	1g 白 淡黄色	96.30, -2.86, 7.65 92.44, -4.50, 24.03

[0046]

[Table 2]
動物繊維の着色結果

	試験片 の繊維	濃色化条件	洗濯		摩擦		汗			
							酸		アルカリ	
			繊維	綿	乾燥	湿潤	繊維	綿	繊維	綿
例 1	絹 羊毛		4-5 4-5	4-5 4-5	4 4-5	4 4	4-5 4-5	4-5 4-5	4-5 4-5	4-5 4-5
	絹	4-ヒドロキシベンゾンズ アルデヒド 2g	4-5	4-5	4-5	4	4-5	4-5	4-5	4-5
	絹 羊毛	トリフルオロ酢酸 30ml	4-5 4-5	4-5 4-5	4-5 4	4 4	4-5 4-5	4-5 4-5	4-5 4-5	4-5 4-5
例 2	絹 羊毛		4-5 4-5							
	絹 羊毛	トリフルオロ酢酸 30ml	4-5 4-5	4-5 4-5	4-5 4-5	4 4	4-5 4-5	4-5 4-5	4-5 4-5	4-5 4-5
例 3	絹 羊毛		4-5 4-5	4-5 4-5	4-5 4-5	4-5 4	4 3-4	4-5 4	4 4	4-5 4
例 4	絹 羊毛		4-5 4-5	4-5 4-5	4 4-5	4 4	4-5 4	4-5 4-5	4-5 4	4-5 4-5
例 5	絹 羊毛		4-5 4-5	4-5 4-5	4-5 4-5	※ 4	4-5 4-5	4-5 4-5	4-5 4-5	4-5 4-5
例 6	絹 羊毛		4-5 4-5	4-5 4-5	4-5 4-5	4-5 4	4-5 4-5	4-5 4-5	4-5 4-5	4-5 4-5
例 7	絹 羊毛		4-5 4-5	4-5 4-5	4-5 4-5	4 4-5	4-5 4-5	4-5 4-5	4-5 4-5	4-5 4-5
例 8	絹 羊毛		4-5 4-5	4-5 4-5	4-5 4-5	※ 4	4-5 4-5	4-5 4-5	4-5 4-5	4-5 4-5
例 9	絹 羊毛		4-5 4-5	4-5 4-5	※ 4-5	※ 4	4-5 4-5	4-5 4-5	4-5 4-5	4-5 4-5
例 10	絹 羊毛		4-5 4	4-5 4-5	4-5 4-5	4-5 3	4-5 3-4	4-5 4-5	4 3	4 4-5
例 11	絹 羊毛		4-5 4-5	4-5 4-5	4-5 4-5	4-5 4	4-5 4-5	4-5 4-5	4 4-5	4-5 4-5

※ 試験実施不可能

[Translation done.]